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(54) Title: HIGH STRETCH ELASTOMERIC PRE-STRECHTED TUBES

(57) Abstract

Elastomeric, low modulus pre-stretched tubular articles supported by removable cores. Embodiments of the articles include those useful for insulators on power cables, and for dielectric stress control of cable splices and terminations. A conductive article is useful for shielding high voltage cables. The articles have low Shore A hardness values and low moduli which allow large expansion ratios and increased wall thicknesses when compared to conventional pre-stretched tubes.

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HIGH STRETCH ELASTOMERIC PRE-STRETCHED TUBES

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Background of the Invention Field of the Invention

The invention relates to a multiplicity of elastomeric articles in the form of pre-stretched tubular members supported on removable cores. The articles may be used to provide insulation, dielectric stress control or shielding for spliced or terminated medium voltage and high voltage power cables. Additionally, the tubular members may be used as sleeves for sealing pipes or conduits.

Description Of The Prior Art

A pre-stretched tube (PST) is a tubular member which is supported in a stretched condition on a support

20 member, usually in the form of an easily removable core. The core of the PST can be external, i.e., on the outside of the tubular member, or it can be inside the tubular member, as is taught in U. S. Patent No. 3,515,798, (Sievert), incorporated herein by reference. It is disclosed that the core is preferably an internal one-piece rigid spiral core having interconnected adjacent coils in a closed helix configuration.

Pre-stretched tubes have several major applications in the electrical industry. They are used in the repair of electrical cables. They are often used to protect spliced connections such that the spliced cable is threaded through a PST carrying support core which has an internal diameter larger than the outside diameter of the cable. With the PST positioned over the cable splice, the plastic core is removed allowing the stretched elastomeric material to recover, approaching its original size, so that the tubing fits snugly over the repaired section of cable.

While the major use of conventional PSTs has been as

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insulating sleeves, certain PSTs may also be used as stress control tubes in a cable termination or spliced cable. Such an application is disclosed in U.S. 3,585,274, (Tomaszewski et al.) which describes a stress 5 control tube composed of high permittivity material containing one or more titanates and/or titanium dioxide. Preferred permittivity levels are disclosed to be at least 5 to 10 times the permittivity of the high voltage cable. U.S. 4,053,702 (Erickson et al.) also discloses a 10 stress control element in the form of a tube which may be slid into place over the end of a terminated cable. The tube comprises a non-rigid elastomeric material. Titanium dioxide is used to provide permittivity levels in the range of from 10 to 25.

U.S. 4,363,842 (Nelson) also discloses the use of high permittivity pre-stretched tubes. It is disclosed that the filler concentration must not be too large or the PST will lose elastomeric characteristics, and fail to fully collapse when the core is removed. This will 20 leave gaps between the power cable and the PST.

The extent of stretching is related to the hardness of the elastomeric material used which is quantified by Shore A hardness values. The current PST materials generally have Shore A hardness values ranging from about 43 to about 60 and are generally limited to a maximum of about a 3 to 1 expansion ratio. Expansion over about 3:1 usually resulted in splitting or tearing of the tube. Consequently, the diameter of the stretched elastomeric tube is limited to about three times the size of the tube in its unstretched condition. Further limitations are imposed by the ability of the core material to support the stretched tube without collapsing prematurely. more the PST is expanded, the greater is the force exerted by the tube as it tries to recover.

35 Consequently, the core must be designed to withstand this force. Where the core is designed for easy removability, there is a trade-off between its wall thickness and its

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ability to collapse on demand. To withstand higher forces, the wall thickness of the core must be increased. This adversely affects removability and cost of the core.

The range of sizes, i.e., external diameters of

spliced cable which may be protected by pre-stretched
tubing, is limited. This limitation results from the
balance which must be maintained between the recovery
force exerted by the stretched elastomeric tube and the
ability of the supporting core to resist crushing. The

range of cable diameters to which PSTs may be applied
could be extended by use of a relatively soft PST with a
high expansion ratio. Therefore, a need exists for a
softer elastomer, suitable for use in pre-stretched
tubes, which possesses an expansion ratio larger than

currently available materials. This higher ratio tubing
could provide a PST for larger diameter cable and
multilayer constructions suitable for high voltage
applications.

Alternatives to single pre-stretched tubes have been disclosed in the art. Two layer sleeves, i.e., sleeves applied as two separate tubes, for termination of electrical cables are disclosed in U.S. 4,714,800, (Atkins et al.). The first tube, applied next to the conductor, is a heat-shrink stress control tube. The second tube is an insulator, the outer facing surface of which is convoluted.

Relatively rigid heat-shrinkable tubular structures are disclosed in U.S. 4,383,131, (Clabburn). The protective sleeve may comprise a conductive outer layer, an insulating inner layer, and optionally a stress grading innermost layer.

Permanently elastic dielectric materials are disclosed in U.S. 4,363,842, (Kehr et al.). The materials are permanently resilient stress control elements which yield at the site of application. The materials are formed into a member which slips over the cable, and not into a pre-stretched tubular member.

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The use of PSTs as insulators in high voltage applications has increased in recent years. Current PSTs have provided effective insulation up to voltage ratings of about 600 volts. There is a need to increase this 5 rating to as high as 5 kilovolts in high voltage applications. Also, there is a need to achieve these high voltage ratings using relatively soft rubbers such as ethylene propylene diene monomer rubber (EPDM). dielectric strength of a selected rubber will indicate 10 whether it is to be used for e.g., insulation or dielectric stress control. It is desirable for insulating materials to have a dielectric strength above about 450 V/mil in high voltage applications. Dielectric stress control requires the use of materials with 15 dielectric strength values in the range of 90-160 V/mil. The higher the dielectric strength of the rubber, the thinner the PST wall can be, which results in less material used and less force on the support core which might cause it to collapse.

Further, the use of pre-stretched tubes as conductive shields for electrical cables has been limited in that, any conductivity is usually lost after expansion and recovery. It would be desirable for certain high voltage applications, to have a pre-stretched tube which would maintain an adequate amount of conductivity after expansion and recovery.

It is, therefore, an object of this invention to provide pre-stretched tubes comprised of low hardness, high expansion ratio elastomeric rubbers, supported on removable plastic cores for use in controlling dielectric stress.

Another object of this invention is to provide elastomeric pre-stretched tubes free from carbon black or containing amounts suitable for use in insulating spliced or terminated medium voltage to high voltage cables.

It has also been discovered that such elastomeric rubbers can be formulated with conductive fillers and

formed into pre-stretched tubes having an expansion of 2 to 3 times the original dimension which will maintain good electrical conductivity and hence good electrical shielding capability even after expansion and recovery.

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Summary of the Invention

The present invention provides a multiplicity of articles comprising elastomeric tubular members supported in a stretched condition on easily removable cores, the tubular members comprising:

- (a) from about 10 to about 60 percent of an oilextended ethylene propylene diene monomer rubber,
- (b) from about 5 to about 25 percent of an elastomer selected from the group consisting of a nonoil-extended ethylene propylene diene monomer rubber and nonoil-extended ethylene propylene rubber,
- (c) from about 5 to about 45 percent of an extender,
- (d) from about 10 to 30 percent of a processing oil,
- (e) from about 2 to about 30 percent of silica,
- (f) from 0 to about 35 percent carbon black, and
- (g) up to about 30 percent of a polymeric plasticizer.

These terms have the following meanings as used herein.

- 1. The term "oil-extended" refers to a rubber in which oil was added during polymerization.
 - 2. The terms "extender" and " nonreinforcing filler" are used interchangeably to mean a non-reactive additive which reduces the amount of primary ingredients necessary in the formulation.
- 3. The term "PST" means pre-stretched tube or prestretched tubular member.

All percents, parts and ratios herein are by weight unless specifically noted otherwise.

One embodiment of the invention provides an insulating PST article having a black tubular member. This article is useful in low voltage and medium voltage cables. Suitable PST materials of this type have a Shore

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A hardness of less than about 33, and an expansion ratio of about 5 to 1. The 300% modulus should be below about 230 pounds per square inch. (1.6 MegaPascal). This PST may contain up to about 35% regular carbon black.

Another embodiment of the invention provides a nonblack or white insulating PST article. Suitable tubular members either contain no conductive fillers, e.g., carbon black, or use conductive fillers in amounts, e.g., less than 1.0 percent which yield insulative 10 compositions useful in medium voltage and high voltage applications. Suitable PST materials of this type have a Shore A hardness of less than about 38, and an expansion ratio of about 5 to 1. The 300% stretch modulus should be below about 300 pounds per square inch (2.1 MPa).

Another embodiment of the invention provides a PST useful for dielectric stress control. Stress control is achieved by incorporating high permittivity fillers into the rubbery polymer composition of the tubular members. Suitable PSTs of this type have a Shore A hardness value 20 of below about 45 and have low moduli, which allows an expansion ratio of about 4 to 1. The 300% modulus should be below about 400 pounds per square inch (2.8 MPa). Furthermore, the elastomeric material has a voltage rating in excess of 600 V. This material is used in IEEE Class 1, 2 and 3 terminations for up to 35 KV rating.

Another embodiment of the invention provides a PST useful for electrical shielding applications. Suitable tubular members contain high amounts of conductive filler, such that even after expansion and recovery, good 30 electrical conductivity is maintained due to interparticle contact. Preferred articles contain at least about 25 percent conductive filler and have a volume resistivity of less than 200 Ω -cm. This PST material has a Shore A hardness of below about 70, and an expansion ratio of about 3 to 1.

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Brief Description of Drawing

Figure 1 is a graph showing the stress-strain behavior of various EPDM rubbers.

Detailed Description of the Invention

The pre-stretched tubes of the present invention have elastic recovery properties whereby the tubular components almost completely recover their original dimensions after the expansion forces are released. rubbers of the present invention have reduced hardness when compared to conventional elastomers having analogous applications, i.e., insulation, stress control, or shielding of medium and high voltage cables and the like. The lower hardness translates into higher expansion 15 ratios, and low moduli, thus allowing the materials to be stretched or expanded to greater ratios than conventional materials.

The materials exhibit good mechanical strength as well as excellent elastic memory such that they remain in a stretched state without fracture over long periods of time, and, when allowed to shrink, recover quickly and almost completely. The material of the present invention may also be manufactured into PSTs and used in a multilayer capacity.

Furthermore, due to the elasticity of the material, one size will cover a greater variety of cable dimensions than most currently available PSTs, without the need for specially reinforced support structures or cores. elastomeric tube is radially expanded, its wall thickness decreases as the diameter of the stretched tube 30 increases.

Another benefit of the softer elastomeric tube is the opportunity to develop combinations of concentric tubular compositions, which may include hard and/or soft elastomers, so as to provide multilayer PSTs with tailored physical characteristics useful in a variety of applications.

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Elastomeric materials used in PSTs need to have high tensile strength, high elongation, and a low modulus. is desirable to exhibit a tensile strength of over 1000 pounds per square inch (6.9 MPa), determined using ASTM 5 D412. For an insulative PST, the elongation should exceed at least about 850%, preferably 900% from the original resting length. For a stress control PST, the elongation should exceed at least about 800% from the original resting length. For a conductive PST, the elongation should exceed at least about 500% from the original resting length.

The modulus of the materials is also important. For an insulative PST useful in low voltage applications, the modulus should be sufficiently low such that less than 15 230 pounds per square inch (psi), (1.6 MPa), is required to elongate the sample 300%. For an insulative PST useful in medium and low voltage applications, the modulus should be such that less than 300 psi (2.1 MPa) is required to elongate the sample 300%. For a stress control PST, the modulus should be such that less than 400 pounds psi (2.8 MPa) is required to elongate the sample 300%. Finally, for a conductive PST, the modulus should be such that less than 1200 psi (8.3 MPa) is required to elongate the sample 300%. All moduli are determined according to ASTM D412.

The material of the present invention will generally be sold as a PST and, thus, should have an expansion ratio of at least about 5, for insulative PSTs, at least about 4 for PSTs used for stress control and at least 3 for shielding PSTs. 30

Elastic recovery is important so the tube fits snugly upon removal of the inner core. A desirable permanent set for all PSTs of the invention is less than about 23%. The large expansion ratio makes possible a 35 greater wall thickness.

The elastomeric material of this invention should contain from about 10 to 60 percent oil-extended ethylene

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propylene diene monomer (EPDM) or an oil-extended ethylene propylene rubber (EPM). More preferably, the invention should contain about 15 to 35 percent of an oil-extended EPDM. Suitable oil-extended rubbers include "Epsyn" P597 from Copolymer Company, and "Polysar" XC-955 available from Polysar Incorporated. These oil-extended EPDM rubbers contain 50 percent rubber solids and 50 percent paraffinic oil or naphthenic oil. Other useful commercially available oil-extended rubbers may contain 10 different amounts/ratios of rubber resin and paraffinic oil, e.g., "Epsyn" P557 and "Epsyn" P558, both available form Copolymer Company, and "Vistalon" 3666, available from Exxon Company.

A nonoil-extended 100 percent solids EPDM or EPM 15 rubber is also used in the amounts of about 5 to about 25 percent, more preferably about 10 to about 12 percent. Any commercially available EPDM or EPM rubber which is nonliquid at room temperature can be used; a preferred rubber is "Nordel" 1470, available from E.I. duPont de Nemours, (DuPont). Liquid EPDM is not recommended for use in compositions of the invention as an unacceptable reduction in elongation is frequently seen.

A processing oil is also added to the mixture to soften the material. There are many commercially available processing oils which would be compatible with the present invention. Preferably, up to about 30 percent of a processing oil is added to the mixture; in the case of a shielding PST, up to about 20 percent is preferred. Oils which may be used include paraffinic petroleum oil, naphthenic petroleum oil, and vegetable or 30 animal derivative oils.

However, increasing the amount of processing oil alone does not create a soft, expandable PST. Rather, processing oil added in large quantities greatly impairs the integrity of the polymer. Unless the ratios of 35 processing oil to rubber are properly chosen and compounded, the resulting material will not completely

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cure. This causes weak mechanical and dielectric strength, poor elastic memory, and poor processibility.

PST articles of the invention may further comprise silica. Silica reinforces the strength of the rubber.

5 Useful silicas include hydrated precipitated silica or fumed silica. The PST article may comprise from about 2 to about 30 percent silica, preferably from about 4 to about 15 percent for insulating compositions. Lower amounts are preferred for stress control (0-10%) and shielding (0-5%) compositions. One source of hydrated silica is "Hisil" 532 EP, available from PPG Industries Inc. Other useful silicas include "Hisil" 233, "Hisil" 210, silicas designated as Cabosil™, available from Cabot Corp., silicas designated as Aerosil™, available from 15 DeGussa, and the like.

extender, or nonreinforcing filler to dilute the rubber, thus increasing the mechanical elongation of the cured composition, as well as providing a cost reduction due to the lesser amount of rubber required. Preferably, the extender comprises from about 5 to about 45 percent of the composition for insulating material. For stress control, the extender is preferably from about 5 to about 15 percent, and for conductive materials, preferably from 0 to about 5 percent. Useful extenders include aluminum trihydrate, clay, talc, kaolin, mica, calcium carbonate, magnesium carbonate, aluminum silicate, magnesium silicate, and the like.

Polymeric plasticizers may also be included in

compositions of the invention. The amount of plasticizer varies with the viscosity of the polymer. Useful plasticizers include polybutenes, such as Indopol™ from AMOCO, and a liquid EPDM, such as Trilene™ available from Uniroyal Chemical. When desired, the useful levels of polymeric plasticizer range up to about 30 percent, preferably 3 to 10 percent.

Insulative and dielectric stress control embodiments

of the present invention may also contain up to about 35 percent carbon black. Carbon black may consist of essentially any commercial grade, including large particulate size thermal types, fine reinforcing furnace grades, and materials termed "conductive carbon black".

A preferred carbon black is a coarse furnace grade (i.e., having an average particle diameter of from about 40 to about 100 nanometers). Carbon black helps reduce dielectric stress by effectively controlling refraction of electrical flux lines associated with cable splices or terminations, yet allows maintenance of a desired level of elasticity.

Where a black, insulative PST for low voltage applications is desirable, the range of carbon black is preferably from about 7 to about 10 percent.

It has now been discovered that even with little or no carbon black, the insulative elastomeric articles of the invention will exhibit a high elongation, good elastic recovery, and high flexibility. Surprisingly, even in compositions of the invention containing less than 1 percent carbon black, the rubber will exhibit no splitting at a 5:1 stretch ratio. Further, the white rubber has higher insulative properties. Pre-stretched tubes using such white rubber are very useful in such applications as high voltage insulators, where conductivity is highly undesirable. Further, nonblack and white compounds are useful where color coding is desirable, and in non-electrical applications, e.g., dental products.

In PSTs useful for dielectric stress control, the preferred range of carbon black is from about 13% to about 30%.

where a conductive PST is desirable for cable shielding, a highly conductive carbon black is required, which has an average particle size well below the range described above. These articles contain at least about 25% conductive carbon black.

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Additives such as antioxidants, fungicides, organic silanes, powdered acrylics, lead oxides, zinc oxide and magnesium oxide may be added to compositions of the invention to vary the properties as desired. Useful adjuvants include natural, surface treated, precipitated and hydrated materials.

The composition can be cured by a curing agent such as a peroxide or sulfur or by exposure to energizing radiation, e.g., ultraviolet radiation, electron beams, radio frequency, microwaves, and the like. The preferred method of cure utilizes a peroxide curing agent.

Possible peroxide curing agents include, but are not limited to dicumyl peroxide, 1,1-di(t-butyl peroxy) cyclohexane, 2,5-dimethyl-2,5-di(t-butylperoxy) hexane-3, ethyl-3,3-di(t-butylperoxy) butyrate, 1,1-bis(t-butylperoxy), 3,3,5-trimethyl cyclohexane, 2,5-dimethyl-1-2,5-bis(t-butylperoxy) hexane, t-butyl cumyl peroxide, a,a'-bis(t-butylperoxy) diisopropylbenzene, n-butyl,4,4-bis(t-butylperoxy)valerate, and the like.

The optimum amount of peroxide depends on the specific peroxide used, the combination of polymeric materials, the acidity of the extenders and the amount of processing oil. Various EPDM rubbers may require different amounts of curing agent to yield the same degree of crosslinking. The most preferred peroxide is α, α' -bis(t-butylperoxy)diisopropylbenzene. The preferred amount is from about 1 to about 5 percent, the most preferred amount is from about 2 to about 3.5 percent.

Pre-stretched tubular members useful for control of dielectric stress further contain up to about 15% of a ceramic filler such as titanium dioxide or barium titanate. Such tubular members also contain up to about 2.5% aluminum flakes.

Articles of the invention are commonly supported by interior cores. The core is made from a tough polymeric material such as flexible cellulose acetate butyrate, polyvinyl chloride or polypropylene. Cores for articles

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of the invention may be formed by spirally wrapping an extruded ribbon and fusing or tacking at spaced points along the ribbon to form a rigid core which supports the PST. The core is thereafter removed by pulling one free end of the ribbon core with sufficient force to separate the ribbon where it is tacked such that it becomes unwound. This permits the cover to relax onto an inserted cable or pipe, which leaves the PST tightly affixed over the cable or pipe.

invention is developed mainly for cable accessory and splicing applications, the materials, because of their excellent expansion ratio as well as elastic recovery, may find many applications in the utility, aerospace, household, sports, games, toys or automotive industries.

other uses for the elastomeric material include environmental protection for screwed cable fittings, knurled or hex nuts on flanges, flared or ferruled fittings, fittings for electrical plugs and cords, bayonet or screwed lamp bases, electrical junctions, sockets or fuses, junctions, splices, or adjoining sleeve fittings for pipes transporting natural gas, water, or other gasses or liquids. Uses also include seals or gaskets, glands, faucets, valves or other mechanical couplings or splices.

When the elastic material of the present invention is used as a sleeve for supporting cables, the sleeve is normally cylindrical both for the ease of manufacture and for maximum utility. Tubular structures of non-uniform diameter are also contemplated as coming within the purview of the invention, a specific example being a conical structure for use as an endcap on a cable termination.

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Test Methods

Elastic Recovery

Elastic recovery is measured by the following method:

- 1) Stabilize an oven at 100° C.
- 2) Cut dumbbells from the sample to be tested using a Die C of ASTM D-412.
 - 3) Mark parallel 1 inch (2.54 cm) bench marks at the approximate center of the sample. (Bench marks must be parallel.)
- 4) Place the dumbbell in the set fixture and stretch the sample until the distance between the bench marks is 2 inches (about 5 cm). This correlates to a 100% strain.
 - 5) Place the loaded set fixture in a 100°C oven for 22 hours.
 - 6) After the 22 hour heat cycle, remove the fixture from the oven and allow the stretched sample to cool at room temperature (21°C \pm 3°C) for 60 minutes.
 - 7) Remove the sample from the fixture and place the sample on a smooth wooden or cardboard surface.

 Release the sample gently.
 - 8) After the sample has been out of the fixture for 30 minutes (\pm 2 minutes), measure and record the distance between the bench marks.
 - 25 9) The calculation is as follows:

The permanent set $% = \frac{100 \text{ (RL-OL)}}{\text{TL-OL}}$

wherein RL = relaxed length (distance between
bench marks after cooling)
OL = original length 1.00 inch (2.54 cm)
TL = test length 2.00 inch (5.08 cm)

35 <u>Physical Properties</u>

The modulus, elongation and tensile strength are measured using ASTM D412. The Shore A Hardness is tested using ASTM D2230. The dielectric strength is tested

using ASTM D149; the dielectric constant is tested using ASTM D150.

Volume Resistivity

The retention of conductivity is a major consideration for a conductive PST. The measurement of volume resistivity of tubing may be performed at various stretch ratios during expansion and at recovery.

at both ends of a tube, which has been painted with conductive silver paint on both ends to reduce contact resistance, and measuring with an ohmmeter. The tubing is then expanded and loaded on a plastic cylindrical rod at a specified diameter to ensure the uniformity of expansion. The rod has copper ends which provide the electrodes of resistivity measurement. The two ends of the tubing must contact the copper portion of the rod. After the measurement, the tubing is removed from the rod and loaded onto a rod having a larger diameter (for expansion) or small diameter (for recovery). The volume resistivity is calculated by the equation

wherein Do is the outside diameter of the tubing sleeve, Di is the inside diameter of the tubing sleeve, R is the measured resistance, and L is the length of the tubing 30 sleeve.

High Voltage Tests

The material to be tested is fabricated into a tubular form. Two tests are run; a 15 KV test and a 25 KV test.

In the 15 KV test, the semiconductor cutback is 9 inches (23 cm.) and the length of the stress control tubular member used is 7.5 inches (19 cm.). The voltage is then applied to the material. This impulse test is

run at both positive and negative polarities. The BIL requirement for 15 KV tubular termination is 110 KV for both positive and negative polarities. The same test sample is then used for an AC step test, where 40 KV is first induced, and increased by 5 KV per 5 minute step.

In the 25 KV test, the semiconductor cutback is 15 inches (38 cm.) and the length of stress control tubular member tested is again 7.5 inches (19 cm.). The BIL test is performed by first running one pre-shot at 110 KV, and then increasing to 150 KV. Again, the test is run at both positive and negative polarities. The BIL requirement for 25 KV tubular termination is 150 KV for both the positive and negative polarities. The same test sample is then used for an AC step test, where 50 KV is first induced, and increased by 5 KV per 5 minute step.

Sample Preparation

The material can be either mill mixed or mixed in a closed mixer such as a Banbury mixer. When mill mixed, 20 it is preferred to use a tight nip roll with cold water running through the rolls to maintain viscosity, improve dispersion and prevent any scorching of the material. Banbury mixing, better viscosity maintenance and improved dispersion may be obtained by maintaining cold temperature in the mixer. This is prefereably achieved by circulating cold water. Generally, carbon black (if used), precipitated silica and nonoil-extended EPDM are added first to obtain better dispersion followed by an oil-extended rubber. After thorough mixing of all ingredients, the curing agent may be added to the 30 mixture. The temperature must be maintained below the decomposition temperature of the curing agent to prevent any premature curing.

The composition can then be formed into a tube using a cold feed extruder equipped with a venting port to help reduce air trapped in the feedstock. The feedstock is extruded as tubing which is then vulcanized, e.g., in an

autoclave using steam. Conventional process aids, process oils, coupling agents, and vulcanizing agents can be included in the compounded elastomeric component.

As mentioned, one of the key characteristics of the present invention is the softness of the material. Black insulating embodiments of the present invention have hardness values of below about 33 Shore A. Shore A hardness is determined according to ASTM D-2240.

The following Table shows Samples A, B, and C which

were similar in all respects except for the amount of oil
in the samples. The properties were measured and it can
be seen that the 300% modulus and the tensile break
strength diminished significantly as the amount of oil
increased. At the same time, the ability of the
elastomer to recover, as indicated by the change in
permanent set, is adversely affected by increasing the
amount of processing oil. If the permanent set values
increase much above about 23%, their value in PST
applications is diminished. The problems observed from
excess oil added to the materials include oil bleeding
out from the rubber, gas generation during curing, and
retardation of the cure system.

	<u>Table 1</u>		
25	Sample A	Sample B	Sample C
Oil Loading, pa per hundred res	rts 70 in (Phr)	120	140
Properties:	. a 46	34	29
Hardness, Shore		300	210
Elongation, %	670	850	930
Tensile @ Break	1578	1295	1056
Permanent Set %	4.0	21	29

The technique used in the present invention remedies the above-mentioned oil problems by the following:

1) using lower volatility plasticizers to

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replace part of the process oil;

- using polymer bound oil which has a higher compatibility level than free oil;
- 3) using more peroxide or coagents to compensate for the effects of the oil. 5

Using more peroxide to improve cure has to be done with caution due to excess peroxide tending to increase the modulus and hardness and reduce the elongation drastically, all of which are not desirable 10 for making a high stretch, low hardness compound.

Example 1 is a typical formulation of a low modulus composition of the invention. The formulation uses an oil-extended EPDM rubber ("Polysar" XC-955), together with a nonoil-extended EPDM rubber ("Nordel" 1470) and the proper selection of other ingredients including process oil, polymeric plasticizer (polybutene), peroxide curative, lead oxide, fungicide, processing aids, antioxidants and coupling agents. formulation, and those included in the other examples are 20 presented using a basis of parts per hundred rubber resin (phr). This represents the combined total of rubber solids present in the composition. Thus, the specified amount of a given component, in any formulation, indicates the quantity to be added for each 100 parts of rubber resin. As indicated previously, where components are described as percentages, the weight of a specified component is used as a basis for measurement. weight percent for e.g., "Polysar XC-955" in Example 1 is 33.6% when stated as a percentage of the total formula weight. 30

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Example 1

	Chemical	Phr	Note
	"Polysar" XC-955	120	Oil-extended EPDM Rubber
•	"Nordel" 1470	40	EPDM Rubber
5	"Hisil" 532 EP	20	Precipitated Hydrated Silica
	"Sunpar" 2280	60	Paraffinic Petroleum Oil
	Zinc Oxide	5	Zinc Oxide
10	"Agerite" White	0.8	Antioxidant
	"Ultranox" 257	1.5	Antioxidant
	"Struktol" WB-16	2	Mixture of Calcium Fatty Acids
	Red Lead	4	Red Lead Oxide, Pb3O4
15	"Furnex" N754	30	Carbon Black
	"Vinyzene" SB-1 ELV	1.5	Fungicide
	"Hydral" 710	45	Aluminum Trihydrate
	"Ucarcil" RC-1	1	Organic Silane
20	"SR-297"	6	1, 3-Butylene Glycol Dimethacrylate
	"Indopol" H-300	10	Polybutene
25	"Vulcup" 40 KE	9.5	α,α'-bis(ter- Butylperoxy)Diisopropyl Benzene on Burgess KE Clay
	TOTAL	356.3	

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The following Table shows a comparison of the low hardness rubber of Example 1 compared to a commercially available PST rubber.

Table 2

The Comparison of Physical Properties of Low

Hardness Rubber (Example 1) with a Typical PST Rubber

5		Low Hardness Rubber	Typical PST
	Properties ·	(Example 1)	Rubber
	Hardness, Shore A	28	46
10	100% Modulus, psi	80	160
	200% Modulus, psi	110	370
	300% Modulus, psi	145	680
	500% Modulus, psi	260	-
	700% Modulus, psi	420	
15	900% Modulus, psi	620	-
	Tensile Strength, psi	1290	1600
	Elongation @ Break	1090	680
	100% Permanent Set, %	13.0	13.0
	Dielec. Strength V/mil	580	400
20	Dielec. Const. @ 1500 V	3.7	5.4
	Dissipation Factor	0.015	0.045

Table 2 shows the properties of the low modulus compound of Example 1 compared to a typical PST compound. The typical PST compound has a Shore A 25 hardness of 46. The low modulus compound has a 28 Shore A hardness. Modulus values listed in Table 2 give a good comparison between extensibility of the The data indicate the force required to two rubbers. stretch a typical PST rubber to 300% is enough to 30 stretch a low modulus rubber more than 900%. The ultimate elongation of the low modulus formulation is about 1.5 times more than the typical PST. If this factor is taken into account, the true tensile strength at the breaking point of the Example 1 35 rubber is greater than the typical PST rubber. The permanent set value demonstrates that the Example 1 rubber has almost the same elastic recovery as a typical PST rubber. The low modulus rubber also

shows better electrical insulating performance as compared to a typical PST rubber. The dielectric strength of Example 1 is above 450 V/mil, which is preferable.

rubbers with various hardnesses. The different curves represent different Shore A hardnesses. The following are the various curves and their associated values.

		Shore A Hardness
10	Curve	SHOLE A HALAHEDE
	A	78
	B	45
	С	35
	D	30
15	E	28
TO	~	•

The figure is a good representation of how easily the material can be elongated. A 78 Shore A rubber is not easily stretched and will break at less than 300% elongation. A 45 Shore A rubber is a typical PST compound. It has a modulus and elongation range that makes it extensible and allows it to be maintained in an expanded shape without breaking. The curves show how the modulus and elongation change with the change of hardness. As the curves indicate, the low hardness rubbers have very low modulus and greater elongation which allows them to be easily expanded to a much greater degree than the high hardness rubbers.

The low modulus, high stretch rubbers exhibit

several outstanding characteristics that are very critical to PSTs. One is the ability to be loaded on a core and remain in a high degree of extension for a long period of time without splitting or breaking apart. Another is substantial elastic recovery after being stretched for a long period of time. Since a PST will be in an expanded state during storage before it is used, it has to be resistant against

splitting on the core or collapsing the core before In order to ascertain whether the tubing it is used. will split or collapse during storage, a test to simulate long-term storage of the PST was developed. The test is performed by expanding the tube radially to a certain degree of extension and thereafter loading the tube on a plastic core. The stretch ratio is determined, for example, by loading a 0.4 inch (1 cm) I.D. tubing on a 2 inch (5.08 cm) O.D. 10 plastic core. The resulting stretch ratio is 4:1, and, for example, if placed on a 2.4 inch (6.1 cm) core, a 5:1 stretch ratio is achieved. The PST is then placed in an oven pre-set at 60°C. The tubing is thereafter constantly inspected to see if 15 splitting has occurred.

The following Table 3 shows the splitting results for the low modulus compound of Example 1.

		Table 3	
20	Characteristics	Regular PST	High stretch PST (Example 1)
	Shore A Hardness	46	29
	Permanent Set %	13	13
25			

Oven Split Test (Number of tubes split out of a total of 4 tubes)

	Stretch Ratio	Number	of Tubes Split
30		Regular PST	Example 1
	3:1	0	0
	4:1	4	0
	5:1	4*	0

*4 Tubes split and/or the cores collapsed

The results from Table 3 indicate regular PST can be stretched to a 3:1 stretch ratio and survive the oven test, at 4:1 the tube splits rapidly, and, if stretched to 5:1, the tube splits or the core

collapses. The high-stretch insulating PST of Example 1 can be stretched beyond about a 5:1 stretch ratio without splitting or collapsing the core due to its high expansion capability and its low modulus.

Also shown in Table 3 is the fact that the highstretch insulative PST of Example 1 has as good a permanent set value as a conventional PST. Permanent set is a measure of elastic recovery of an elastomeric material. The permanent set is a very 10 important property and indicates the final dimensions of the tubing after the core is removed. The tubing will often be subjected to high stretch (e.g., 5:1) and stored for long periods of time. Thus, to ensure a proper fit of the PST after removal of the support 15 core, the permanent set should not exceed 23%.

Comparative examples 2C and 3C show formulations omitting a nonoil-extended EPDM rubber. properties of comparative examples 2C and 3C are not satisfactory for low-hardness rubbers. Comparative example 3 meets every requirement with the exception of a high permanent set. Comparative example 2C shows excellent elastic recovery, but insufficient elongation.

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			2 •	
	<u>Ex</u> <u>Chemical</u>	eample 2C Phr	Example Phr	3C Note
	"Polysar" XC-955	200	-	Oil-extended EPDM Rubber
5	"Epsyn" N997	-	200	Oil-extended EPDM Rubber
	"Statex" N550	30	40	Carbon Black
10	"Hisil" 532 EP	35	25	Precipitated Hydrated Silica
	"Sunpar" 2280	50	30	Paraffinic Petroleum Oil
15	Zinc Oxide	5	5	Zinc Oxide
	"Agerite" MA	1.25	1.25	Polymerized 1,2-Dihydro- 2,2,4-trimethylquinoline
20	"Factice"	20	. 20	Vulcanized Vegetable Oil
	"struktol" WB-16	2	. 2	Mixture of Calcium Fatty Acids
25	"TRD" 90	4	4	Red Lead
	"Ucarcil" RC-1	1	1	Organic Silane
30	"SR-297"	8	8	1,3-Butylene Glycol Dimethacrylate
	"Dicup" 40 KE	16	0	Dicumyl Peroxide on Burgess KE Clay
35	"Vulcup" 40 KE	0	10	α,α'-bis(ter- Butylperoxy)Diisopropyl Benzene on Burgess KE Clay
40	TOTALS	372.25	346.25	
40	Properti	<u>es</u>	Exar	mple 2C Example 3C
	Hardness, Shore	A		30 28
	100% Modulus, ps	si		66 79
	200% Modulus, pa	si		12.3
4!				233 202
	Tensile Strengt		1	065 1387
	Elongation @ Br			821 881
	100% Permanent			15.1 31.3
	Dielec. Streng			448 256
5	O Dielec. Consta			4.1 5.3
J	Dissipation Fac			0.024 0.036

As shown in the following Examples 4 and 5, by using a different proportion of the nonoil-extended EPDM polymer and the oil-extended EPDM polymer from Example 1, the desired properties of the low modulus rubber can be achieved by balancing the amount of silica, carbon black, processing oil and polymeric plasticizer. Both Examples 4 and 5 exhibit low modulus, low hardness, high elongation and good elastic recovery properties. Example 5 contains just a trace of carbon black which is used as a coloring compound. A non-black, low hardness rubber can be made by replacing the carbon black with precipitated hydrated silica.

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	Chemical	Example 4 Phr	Example Phr	5 Note
_	"Polysar" XC-955	50	80	Oil-extended EPDM Rubber
5	"Nordel" 1470	· 75	60	EPDM Rubber
	"Furnex" N754	30	0.3	Carbon Black
10	"Hisil" 532EP	20	45	Precipitated Hydrated Silica
	"Sunpar" 2280	65	60	Paraffinic Petroleum Oil
15	Zinc Oxide	5	5	Zinc Oxide
	"PLX" 841	1	1	Antioxidant Blend
20	"Ultranox" 257	1.5	1.5	Antioxidant
20	Red Lead	4	3	Red Lead Pb ₃ O ₄
25	"Struktol" WB-16	2	4	Mixture Oxide of Calcium Fatty Acids
23	"Vinyzene" SB-1 ELV	1.5	1.5	Fungicide
30	"Hydral" 710	40	60	Aluminum Trihydrate
30	"Ucarcil" RC-1	1	1	Organic Silane
35	"SR-297"	6	6	1,3-Butylene Glycol Dimethacrylate
33	"Indopol" H-300	20	15	Polybutene
40	"Vulcup" 40 KE	9	9	α,α'-bis(ter- Butylperoxy)Diisopropyl Benzene on Burgess KE Clay
	TOTALS	331	352.3	

	Properties	Example 4	Example 5
	Hardness, Shore A	30	30
	100% Modulus, psi	83	70
	200% Modulus, psi	125	105
5	300% Modulus, psi	180	140
5	500% Modulus, psi	_	250
	700% Modulus, psi	-	410
	Tensile Strength, psi	1250	1190
	Elongation @ Break	1028	1000
	100% Permanent Set, %	12.9	12.9
10	Dielec. Strength V/mil	537	487
	Dielec. Constant @ 1500 V	3.4	2.5
	Dissipation Factor	0.018	0.017

15 <u>Example 6</u>

Non-Black Pre-Stretched Tubular Membrane

	Chemical Pa	rt (phr)	Note
	"Polysar" XC-955	200	Oil Extended EPDM
20	"Agerite Stalite-S"	3	Mixture of Octylated Diphenylamines
	"Vanox" ZMTI	1	Zinc 2- mercaptotolylimidazole
	"TE-80"	2	Fatty Acid Mixtures
	"Ucarsil" RC-1	1.5	Organofunctional Silane
25	"Burgess" CB	50	Treated Aluminum Silicates
	"Hisil" 532 EP	40	Precipitated Hydrated Silica
	Zinc Oxide	5	Zinc Oxide
30	TRD-90	4	Red Lead Dispersion
	"Indopol" H-100	45	Polybutene
	"Microcheck" 11-D	1.5	Fungicide
	"Drimix" SR297-75E	6.5	1,3 Butylene Glycol Dimethacrylate
35	"Vulcup" 40 KE	7.5	α,α'-bis(ter- Butylperoxy)Diisopropyl Benzene on Burgess KE Clay

40 TOTAL 367

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	Physical properties: .	
	100% Modulus	66 psi
	200% Modulús	98 psi
	300% Modulus	156 psi
5	Tensile Strength	1022 psi
	Ult. Elongation	949%
,	Hardness, Shore A	28
	Permanent Set, 22 hr. @ 100°C	11.7%
10	Dielec. Strength V/mil	610
	Dielec. Constant	2.91
	Dissipation Factor @ 23°C, 1200 Volt	0.0092

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Example 7
Stress Control PST with High Permittivity

			•
	Chemical	Part (phr)	<u>Note</u>
	"Epsyn" 7506	40	EPDM Rubber
5	"Polysar" XC-955	120	Oil Extended EPDM
	"PLX 854"	1.5	Antioxidant Blend
10	"Ultranox" 257	1.5	Phenol, 4-methyl, Reaction Products with Dicyclopentadiene and Isobutylene
10	"Ucarsil" RC-1	1	Organofunctional Silane
	Zinc Oxide	5	Zinc Oxide
	"Tipure" R902	15	Titanium Dioxide
	"Sunpar" 2280	30	Paraffinic Oil
15	"Indopol" H-300	5	Polybutene
	"Statex" N550	35	Carbon Black
	"Furnex" N754	45	Carbon Black
	"Ticon" P	30	Barium Titanate
	"WB-16"	2	Fatty Acid Mixture
20	"Silberline" DF	1651 9.4	Aluminum Flakes
	"Drimix" SR-197-	75E 5	1,3 Butylene Glycol Dimethacrylate
25	"Vulcup" 40 KE	7.6	α, α'-bis(ter- Butylperoxy)Diisopropyl Benzene on Burgess KE clay

TOTAL 353

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	Property	Example 7
	100% Modulus, psi	133
	200% Modulus, psi	231
	300% Modulus. psi	389
5	Tensile Strength, psi	1735
-	Ult. Elongation, %	835
	Hardness, Shore A	42
10	Permanent Set, 22 hr. @ 100°C 100%/30 Min. Recovery, %	14.1
	Dielec. Strength V/mil	105
	Dielec. Constant Dissipation Factor @ 23°C, 1200 Volt	0.11

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The high voltage test results for Example 7 are shown in below. Example 7A is a test on a class 3 termination with Example 7 applied as the stress control component at 15 KV voltage rating with 9 inch (23 cm.) cutback of the semiconductor layer of the tube. Example 7B shows a 25 KV voltage rating with 15 inch (38 cm.) cutback of the semiconductor layer of the tube.

Example 7A

		_
25	<u>Test</u>	Result
	Partial Discharge CSV (KV-RMS) CEV (KV-RMS)	50 -
30	BIL Surges KV-Crest KV-Crest	+113 -111
35	5 minute AC Step Test KV-RMS	80
40	Maximum Impulse KV-Crest KV-Crest	+170 -171

	Test	Example 7B	Result
5	Partial Discharge CSV (KV-RMS) CEV (KV-RMS)		46 41
10	BIL Surges KV-Crest KV-Crest		+152 -152
	5 minute AC Step Test KV-RMS		90
15	Maximum Impulse KV-Crest KV-Crest		+188 -173

As can be seen, the composition of the invention passed both the 15 KV and 25 KV testing. Examples 8-10 demonstrate conductive PST materials useful for electric shielding.

25

Example 8

		<u></u>	
	Chemical	Part (phr)	Note
30	"Nordel" 1470	40	EPDM Rubber
	"Polysar" XC-955	120	EPDM Rubber, 50% Oil Extended
	"Agerite MA"	1.5	Antioxidant Blend
	"Ucarsil" RC-1	1.5	Organofunctional Silane
35	Zinc Oxide	3	Zinc Oxide
	"Sunpar" 2280	55	Paraffinic Oil
	"Conductex" 975	75	Conductive Carbon Black
	"Ketjen" EC-300J	26	Conductive Carbon Black
40	"SR-297"	3	1,3 Butylene Glycol Dimethacrylate
	"Vulcup" 40 KE	9.1	α,α'-bis(ter- Butylperoxy)Diisopropyl Benzene on Burgess KE Clay
45	መ⊜መሽፒ.	334.1	

TOTAL 334.1

Total Carbon Black, 30.7%

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Physical properties:

100% Modulus 246 psi
300% Modulus 1023 psi
Tensile Strength 2116 psi
Ult. Elongation 566%
Hardness, Shore A 61
Permanent Set 17%
Volume Resistivity 21 ohm.cm

Conductivity after recovery for a sample 10 according to example 8 was tested. The original volume resistivity of the tubing was 41.8 ohm-cm (as opposed to the slab sample tested above at 21 ohm-The volume resistivity increased when the sample was stretched to 150% and then decreased at 280% stretch. During the recovery, volume resistivity increased when the tube recovered to 176% stretch ratio and then decreased when it was recovered further. In all, the original low volume resistivity maintains a low value throughout the entire process of expansion and recovery. As can be seen for this particular rubber the volume resistivity never goes beyond 150 ohm-cm under the influence of mechanical stress and strain.

25

		Expans	sion	Recove	ery.	
	<u>Original</u>	150%	280%	<u>175%</u>	140%	
Volume Resist.	42	100	61	147	133	

Comparative Example 9C

	Chemical Pa	rt (phr)	Note
	"Trilene" 65	50	Liquid EPDM Rubber
5	"Polysar" XC-955	100	EPDM Rubber, 50% Oil Extended
•	"Agerite" MA	1.5	Antioxidant
	"Ucarsil" RC-1	1.5	Organofunctional Silane
	Zinc Oxide	3	Zinc Oxide
	"Sunpar" 2280	65	Paraffinic Oil
10	"Carbowax" PEG 3350	1.5	Polyethylene Glycol
10	"Conductex" 975	65	Conductive Carbon Black
	"Ketjen" EC-300J	30.	Conductive Carbon Black
	"SR-297"	3	1,3 Butylene Glycol Dimethacrylate
15	"Vulcup" 40 KE	8.5	α,α'-bis(ter- Butylperoxy)Diisopropyl Benzene on Burgess KE

TOTAL 264

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Total Carbon Black, wt% 36.0%

Physical properties:

	100% Modulus	737 psi
25	300% Modulus	-
	Tensile Strength	1184 psi
	Ult. Elongation	162%
	Hardness, Shore A	77
	Permanent Set	20.4%
30	Volume Resistivity	8 ohm-cm

As can be seen in the above example, the use of liquid EPDM may result in generally good physical characteristics. However, the ultimate elongation is unacceptably low.

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Example 10

	Chemical	Part (phr)	Note
	"Polysar" XC-955	200	EPDM Rubber, 50% Oil Extended
5	"Agerite" MA	1.5	Antioxidant
•	"Ucarsil" RC-1	2	Organofunctional Silane
•	Zinc Oxide	5	Zinc Oxide
•	"Sunpar" 2280	35	Paraffinic Oil
	"Conductex" 975	100	Conductive Carbon Black
10	"Ketjen" EC-300J	35	Conductive Carbon Black
-	"SR-297"	3	1,3 Butylene Glycol Dimethacrylate
15	"Vulcup" 40 KE	8.7	α,α'-bis(ter- Butylperoxy)Diisopropyl Benzene on Burgess KE Clay
	TOTAL	390.2	_

Total Carbon Black, wt% 34.6%

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Physical properties:

	100% Modulus	293 psi
300% Modulus		1124 psi
	Tensile Strength	1698 psi
25	Ult. Elongation	500%
	Hardness, Shore A	69
	Permanent Set	28.3%
	Volume Resistivity	11 ohm-cm

The scope of the invention is not limited to the specific details set forth herein for purposes of illustration, but is solely defined by the claims. Various modifications are possible without departing from the spirit of the invention.

What is Claimed is:

- An article comprising an elastomeric tubular member supported in a stretched condition on an easily removable core, said tubular member comprising:
 - a) from about 10 to about 60 percent of an oilextended ethylene propylene diene monomer rubber,
- b) from about 5 to about 25 percent of an elastomer selected from the group consisting of nonoil-extended ethylene propylene diene monomer rubber and nonoil-extended ethylene propylene rubber,
- c) from about 5 to about 45 percent of an extender,
 - d) from about 10 to about 30 percent of a processing oil,
 - e) from about 2 to about 30 percent of silica, and
 - f) from 0 to about 30 percent of a polymeric plasticizer.
- 2. The article of claim 1 further containing 25 about 1 to 5 percent of peroxide as a curing agent.
 - 3. The article of claim 1 having a permanent set of less than about 23%.
- 4. The article of claim 1 wherein said extender is selected from the group consisting of aluminum trihydrate and aluminum silicate.
- 5. An insulative article useful in low voltage applications comprising the elastomeric tubular member of claim 1 further containing up to about 35 percent carbon black, said tubular member having a

Shore A hardness value of less than about 33, a 300% stretch modulus below about 230 pounds per square inch, an elongation of at least about 850%, and an expansion ratio of about 5 to 1.

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- 6. The article of claim 5 wherein said article contains from about 7 to about 10 percent carbon black.
- 7. The article of claim 5 further containing about 1 to 5 percent of peroxide as a curing agent.
 - 8. The article of claim 5 having a dielectric strength above about 450 V/mil.

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- 9. A nonblack insulative article useful in medium and high voltage applications comprising the elastomeric tubular member of claim 1, containing less than about 1% carbon black, wherein said tubular member has a Shore hardness value of less than about 38, a 300% stretch modulus below about 300 pounds per square inch, an elongation of at least about 850%, and an expansion ratio of about 5 to 1.
- 25 10. An article useful for dielectric stress control comprising an elastomeric tubular member supported in a stretched condition on an easily removable core, said tubular member comprising:
- a) from about 10 to about 60 percent of an oilextended ethylene propylene diene monomer
 rubber,
 - b) from about 5 to 25 percent of an elastomer selected from the group consisting of a nonoil-extended ethylene propylene diene monomer rubber and nonoil-extended ethylene propylene rubber,
 - c) from about 5 to about 15 percent of an extender,

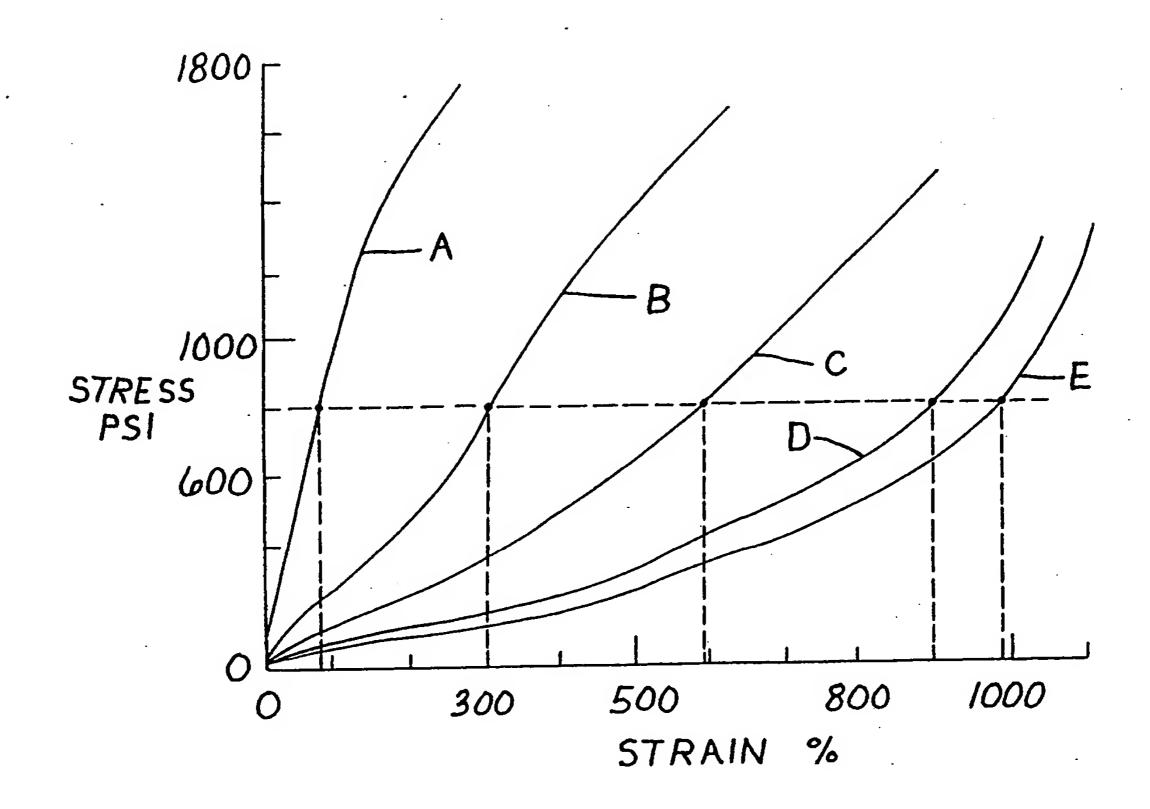
- d) from about 10 to about 30 percent of a processing oil,
- e) from 0 to about 10 percent of silica,
- f) from about 10 to about 35 percent carbon
- 5 black,
 - g) from 1% up to about 15% of a ceramic filler selected from titanium dioxide and barium titanate, and
- h) from 0 to about 30 percent of a polymeric plasticizer, wherein said tubular member has a Shore A hardness value of below about 45, an elongation of at least about 800%, a 300% stretch modulus below about 400 pounds per square inch, and an expansion ratio of about 4 to 1.
 - 11. An article according to claim 10 having a dielectric strength of from about 90 to about 160 V/mil.

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- 12. An article according to claim 10 further comprising from about 1% to about 3% aluminum flakes.
- 13. The article of claim 10 further containing about 1 to about 5 percent of peroxide as a curing agent.
- 14. A conductive article useful for shielding high voltage cables comprising an elastomeric tubular member supported in a stretched condition on an easily removable core, said tubular member comprising:
 - a) from about 10 to about 60 percent of an oilextended ethylene propylene diene monomer rubber,
 - b) from about 5 to about 25 percent of an elastomer selected from the group consisting of

- a nonoil-extended ethylene propylene diene monomer rubber and nonoil-extended ethylene propylene rubber,
- c) from 0 to about 5 percent of an extender,
- d) from 0 to about 20 percent of a processing oil,
 - e) from 0 to about 5 percent of silica,
 - f) at least about 25 percent of a highly conductive carbon black, and
- 10 h) from 0 to about 15 percent of a polymeric plasticizer, wherein said tubular member has a shore A hardness value of below about 70, an elongation of at least about 500%, a 300% stretch modulus below about 1200 pounds per square inch, and an expansion ratio of about 3 to 1.
 - 15. The article of claim 14 further containing about 1 to about 5 percent of peroxide as a curing 20 agent.
 - 16. The article of claim 14 having a volume resistivity of less than 200 ohm-cm.



International Application No

		tional Application	
CLASSIFICA	TION OF SUBJECT MATTER (if several classification a	selfication and IPC	
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	FACTURING COMPANY) 09 September 1981 (09.09.81),	
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	see claims; page 3, - page 4, line 3.		
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ANHANG

zum internationalen Recherchenbericht über die internationale Patentanmeldung Nr.

ANNEX

to the International Search Report to the International Patent Application No.

ANNEXE

au rapport de recherche international relatif à la demande de brevet international n*

PCT/US91/02422 SAE 47005

in diesem Anhang sind die Mitglieder der Patentfamilien der im obengenannten internationalen Recherchenbericht cited in the above-mentioned interangeführten Patentdokumente angegeben. Diese Angaben dienen nur zur Unterrichtung und erfolgen ohne Gewähr.

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La présente annexe indique les membres de la famille de brevets relatifs aux documents de brevets cités dans le rapport de recherche international visée ci-dessus. Les reseignements fournis sont donnés á titre indicatif et n'engagent pas la responsibilité de l'Office.

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